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Mechanical Properties of Polypropylene/Epoxydized Natural Rubber Blend via Mixing Ratio Analysis

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Abstract-This research is to investigate the effect of mixing ratio to the properties of polypropylene (PP) when incorporated with epoxydized natural rubber (ENR). The PP/ENR blend was prepared by melt compounding using an internal mixer and vulcanized through sulfur curing. Mechanical testing such as tensile test, hardness test and impact test was performed to characterize the properties of PP/ENR blend. It was clearly observed that ENR percentage in PP/ENR blend increases the toughness and flexibility of the samples. If compared to pure PP, 40/60 PP/ENR blend showed improvement of elongation to break and impact strength up to 68% and 56%, respectively. In contrast the tensile strength and hardness decreases as the amount of PP decreases. It was contributed by properties imparted by elastic chains of cross-linked ENR. The obtained properties showed good correlation with fracture surfaces observed in microscopy analysis performed by Field Emission Scanning Electron Microscope at magnification of 500- and 5000x.

Keyword-Polypropylene, Epoxydized Natural Rubber, Blend, Mechanical properties

I. INTRODUCTION

Thermoplastic elastomers (TPEs) belong to a class of material that combines physical properties of thermoplastic and elastomer. They exhibit properties typical of rubber materials but can be processed like thermoplastics. Blending of polymers is a common technology, frequently applied in order to develop a product with superior mechanical properties from inexpensive polymer material (Zurina 2006). It is aimed to obtain materials, which as far as possible combine the advantages, but not their disadvantages. Furthermore, TPEs give better material utilization than thermosetting materials because scraps and rejects can be recycled and re-worked (Ismail et al. 2001).

Thermoplastic elastomer based on natural rubber and thermoplastic blends are classified as 'thermoplastic natural rubber' (TPNR) blends. There are two types of TPNRs; 1) prepared by blending NR with thermoplastic such as polyolefin to get co-continuous phase morphology

and it is technologically classified as 'thermoplastic polyolefin (TPO)', 2) It is known as 'thermoplastic vulcanizate (TPV)' which prepared by blending NR with polyolefins and the rubber phase is vulcanized during the mixing process at high temperature; the process is known as 'dynamic vulcanization (DV)' (Nakason et al. 2006).

Polypropylene (PP) is an additional polymer with a large molecular weight distribution (Meyer and Keurentjes 2005). There are three types of PP such as atactic (aPP), isotactic (iPP) and syndiotactic (sPP) (Byrdson 1995; 1999). It has wide range of application due to its unique properties such as high melting temperature, low density, high chemical resistance, and resistance to heat. On the other hand, PP exhibits poor impact strength which gives limitation to several other applications (da Costa et al. 2010; Hogt et al. 1997; Utacki 1994).

Epoxydized natural rubber (ENR) is a material of great interest, exhibiting a double functionality for crosslinking (double bonds and epoxy site) while retaining most of the properties of natural rubber (Pire et al. 2010). The epoxydation of natural rubber can be performed using peracid; generated from reaction of formic acid and hydrogen peroxide (Liau 1998). The properties of ENR are gradually changed with increasing degree of epoxydation (Myriam et al. 2010; Liau 1998). Presence of epoxy groups in rubber chain imparted great properties to natural rubber such as oil resistance, low gas permeability, good wet grip and high damping characteristics (Mohamad et al. 2008; 2010; Thitthammawong et al. 2007).

The main objective of blending elastomeric phase (ENR) into PP is to improve its flexibility and behaviour at low temperatures (Lopez-Manchado et al. 2001). In polymer blends with a crystallizable component, the final properties are determined by: (1) mode and state of dispersion of rubbery domains in the crystalline matrix; (2) the texture, dimensions and size distribution of spherulites of the matrix; (3) the inner structure of spherulites, i.e. lamellar and inter lamellar thickness; (4) physical structure of inter spherulitic boundary regions and amorphous inter

lamellar regions; and (5) the adhesion between the rubbery domains and the crystalline matrix (George et al. 2000). In a study by Koshy et al. (re-stated in George et al. 2000); the crystallinity of the blends decreased with the increase in NR content, and the interplanar distance, d values increased on the addition of NR indicating the migration of NR phase into the interchain space of EVA. Blends of polypropylene with nitrile rubber possess the excellent processing characteristics and mechanical properties of polypropylene with the oil resistance and flexibility of NBR.

Studies conducted on PP/ENR or ENR/PP blend were focusing more on studying the effect of dynamic vulcanization (Nakason et al. 2006; 2008; Thithamwong et al. 2007) or irradiation (Senna et al. 2008) to the properties of the blend. There is less significant effort on studying the effect of PP: ENR ratio to its physical or mechanical properties. In this paper, we have investigated the effect of mixing ratio between thermoplastic and rubber material to mechanical properties of polypropylene/epoxidized natural rubber blends. The properties were supported by morphology analysis on the tensile fracture surfaces.

II. EXPERIMENTAL

A. Material

PP used in this research is Polypropylene homopolymer TITANPRO 6531 (isotactic type) with a specified melt flow index of 3.5 g/10 minutes. It was supplied by Titan PP Polymers (M) Sdn. Bhd. ENR was supplied by the Malaysian Rubber Board under the trade name of ENR 50 with 53% epoxidization. The average Mooney viscosity [measured at ML (1+4) 100°C] was 85.5, and the average specific gravity at approximately 25°C was 0.9366. Sulfur was used as vulcanizing agent whereas zinc oxide and stearic acid were used as activators in the sulfur curing system. The sulfur, zinc oxide, and stearic acid were purchased from average specific gravity at approximately 25°C was 0.9366. Sulfur was used as vulcanizing agent whereas zinc oxide and stearic acid were used as activators in the sulfur curing system. The sulfur, zinc oxide, and stearic acid were purchased from Sin Rubtech[®].

B. Mixing and preparation of sample

The formulation of PP/ENR blends is given in Table 1. The dynamic vulcanization was carried out using a Haake RHEOMIX OS internal mixer. Mixing chamber was 60 cm³ and the batch sizes were 50 ± 5g. The mixer was operated at a constant rotor speed between 60 rpm-80 rpm at temperature range of 170°C - 200°C. The mixing for each batch took place for 10 minutes: firstly, PP and ENR was added into the mixing chamber and mixed for 8 minutes, then, sulfur was added and mixing continued for another 2 minutes to complete the dynamic vulcanization.

The produced TPV was immediately removed from the chamber and left at room temperature for 24 hours. Then, samples were pressed using GT7014-A hot press from Gotech about 5 minutes at temperature of 185°C and pressure of 1800kg/cm². Subsequently, the samples were cooled down under pressure to room temperature for 3 minutes. Samples were cut into desirable sizes according to ASTM standards of various mechanical testing. It was then kept at room temperature for 24 hours before further testing.

Table 1. Formulation of PP/ENR blend

Materials/Chemicals	(phr)	(gram)
PP/ENR		
▪ 70/30 PP/ENR	100.0	45.66
▪ 40/60 PP/ENR		
Zinc oxide	5.0	2.28
Stearic Acid	2.5	1.14
Sulfur	2.0	0.91
Total	109.5	50.00

C. Mechanical properties

Tensile test was carried out according to ASTM D638. It is the most common plastic strength specifications and covers the tensile properties of unreinforced and reinforced plastics. This test method using standard "dumbbell" or "dogbone" shaped specimens at 3 mm thickness. Dumbbell specimens of PP/ENR blends were cut from moulded sheets using a Gotech cutter machine. The tensile test was performed using Autograph AG-IC Universal Testing Machine from Shimadzu Scientific Instruments at a cross head speed of 2.0 mm/min at room temperature, 25 ± 5°C.

Hardness of the samples was tested using a Shore Type D CV durometer according to ASTM D2240. The standard test specimen for ASTM D2240 is 64 x 12.7 x 3.2 mm (2½ x ½ x 1/8 inch). The most common specimen thickness is 3.2 mm (0.125 inch), but the preferred thickness is 6.4 mm (0.25 inch) because it is not as likely to bend or crush.

Samples prepared according to ASTM D 256 were tested for impact strength for Izod pendulum impact resistance using an impact tester. Un-notched specimen was held as a vertical cantilevered beam and impacted by a pendulum.

III. RESULTS & DISCUSSIONS

A. Mechanical Properties

Figure 1 and 2 shows the comparison of tensile strength and Young's modulus between pure PP and PP/ENR blends. It can be seen that the tensile strength and Young's modulus decreases with increasing rubber content in the blend. These reductions were due to the decreased in blend rigidity with increasing ENR content. At lower rubber content (below 50%), the elastomer phases remained as dispersed bodies. As the rubber content increases further,

particle–particle interaction of the rubber phases hence occlusion and accounts for the observed decrease in tensile strength and Young's modulus of PP/ENR blends. In PP/ENR blends, the molecular entanglements in the rubber chains prevent rapid flow and disturbed the ability of the PP to move (Jefferie et al. 2012) and to be strain-crystallized in response to the applied stress.

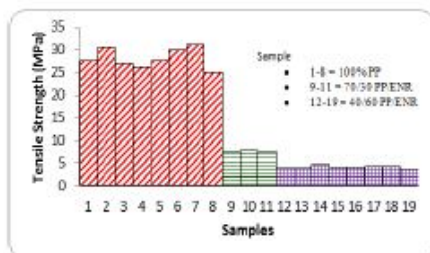


Figure 1 Tensile strength vs sample's number

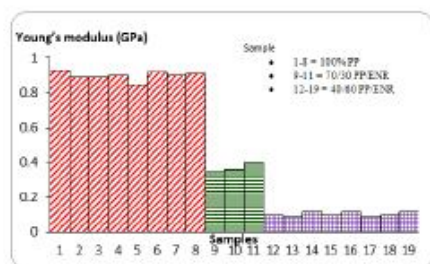


Figure 2 Young's Modulus vs sample's number

According to George et al. (2000), rubber particles are present in inter- and intra-spherulitic region of the crystalline phase plastic (Figure 3). Hence the observed decrease in crystallinity is due to the fact that the formation of crystallites in the blend was affected by the presence of rubber particles. This results in the lower tensile strength of the PP/ENR blend if compared to virgin PP. Furthermore, the presence of crosslinking in thermoplastic matrix limits the flow and mobility which attributes to the rigidity of the polymeric chains of the 70/30 PP/ENR blend; increase the stiffness of the blend (Zurina et al. 2006). In contrast, the crosslinking in rubber matrix of 40/60 PP/ENR blend impart elastic behaviour and lowers the Young's modulus of PP due to rubber-like properties introduced by the ENR (Senna et al. 2008).

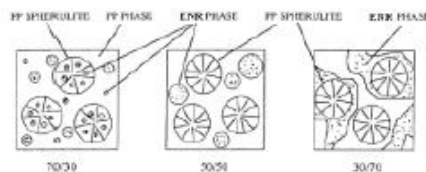


Figure 3 Proposed schematic representation of the crystalline structure of binary PP/ENR blends (Adapted from George et al. 2000)

As illustrated in Figure 4, PP/ENR 40/60 had the highest value of elongation to break (EB) if compared to pure PP and 70/30 PP/ENR. It depicts the better elasticity, toughness and flexibility of the material. The pure PP shows the lowest value of EB whereas, 70/30 PP/ENR shows moderate value in between pure PP and 40/60 PP/ENR. The EB value increases with increasing ratio of ENR in thermoplastics (Senna et al., 2008). It is directly represents the improvement on the ability of the material to absorb energy as rubber content increases since crosslinking in rubber phases (ENR-50) will impart the elastic behavior to the blend (Zurina et al. 2006). Besides, the 30 wt% addition of ENR into PP increased the inter-planar distance (d value) which indicates that rubber particles are present in the intra-spherulitic structure of PP (George et al. 2000). As the concentration of ENR increased to 50 wt% and above the d value decreased resulted from occlusion of rubber particles in inter spherulitic regions due to the large size of ENR at higher concentrations.

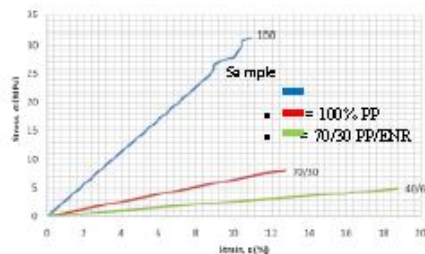


Figure 4 Stress vs strain of pure PP, 70/30 PP/ENR blend and 40/60 PP/ENR blend

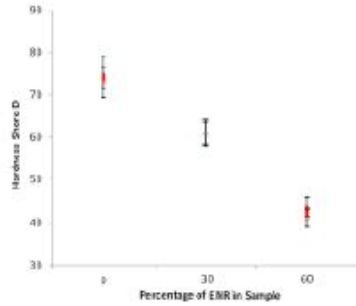


Figure 5 Hardness vs rubber content in PP/ENR blend

Figure 5 shows the hardness versus rubber content in PP/ENR blend. The pure PP (0 wt% of ENR) shows the highest hardness in the range of 73 to 76 Shore D if compared to 40/60 PP/ENR and 70/30 PP/ENR. Hardness indirectly represents the stiffness or rigidity of the material which measures the ability of the material to be scratched or indented by other material. The stiffness decreases as the rubber content increases in the sample. It was clearly observed in 40/60 PP/ENR samples. The samples show the lowest hardness in the range of 40 to 44 Shore D which indicates reduction of about 44 % if compared to pure PP. This is highly contributed by elastic properties of rubber phase in the blend. It shows good agreement with the EB values obtained in tensile test (Figure 4).

Figure 6 shows the impact strength of PP/ENR blends in comparison with pure PP. The 40/60 PP/ENR shows the highest impact strength in the range of 2.1 to 2.7 J/m if compared to pure PP and 70/30 PP/ENR. Higher impact strength represents a higher resistance of the material to fracture under impact loading. This is related to toughness of the material whereby it measures the ability of the material to withstand both plastic and elastic deformations. It depicts the amount of energy that required by the material to break the bond before fractured. It was in agreement with results observed in tensile test whereby EB values increases as the ENR content increases. In this case, ENR is tougher than PP and make it a good candidate to increase the flexibility of thermoplastic material. However the impact strength of 70/30 PP/ENR deviated from this correlation as it shows the lowest impact strength (in the range 0.9 to 1.0 J/m) if compared to virgin PP or 40/60 PP/ENR. Localized and catastrophic deformation in impact test lowers the ability of rubber particles in intra-spherulitic structure of PP to absorb energy, efficiently. These caught rubbers act as frozen bodies and initiate brittle failure of the blend. It concludes that energy absorption in a tensile test cannot be correlated with the level of toughness at impact strain rate (Chen and Evans 2008; Mohamad et al. 2011).

Table 2 summarizes the tensile properties, hardness and impact strength of PP/ENR blends. Tensile strength and

EB decrease as the ENR loading increase. On the other hand, Young's modulus also decreases with increasing of ENR in the blends. In terms of hardness, increases of rubber decreases the hardness of PP/ENR blends. While, increased of ENR higher than 50 wt% increased the impact strength of PP/ENR.

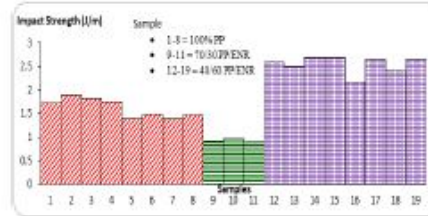


Figure 6 Impact vs rubber content

Table 2 : Mechanical properties of ENR filled various loading of PP/ENR blends

Properties	Type of sample		
	100 PP	70/30 PP/ENR	40/60 PP/ENR
Tensile strength (MPa)	28.31	7.78	4.25
Young's Modulus (MPa)	0.89	0.37	0.105
Elongation at Break (%)	9.89	12.37	16.60
Hardness (Shore D)	74.33	60.93	42.39
Impact Strength (J/m)	1.63	0.93	2.55

B. Morphological Analysis

Figure 7 and 8 shows the tensile fracture surfaces of PP, 70/30 PP/ENR and 40/60 PP/ENR blends. The dark phases represent the ENR and the bright phases correspond to the PP. Most of the fracture surfaces show spherical shaped dimples from pulled-out of PP domains or ENR domains except for the fractograph of unfilled PP, as it can be seen in Figure 7(a) and Figure 8(a). The fractograph of the unfilled PP shows characteristics of ductile fracture under uniaxial tensile loads with the obvious pattern of shear yielding on the surface. In Figure 7(b) and 8(b), fracture surface of 70/30 PP/ENR blend reveals that the ENR were dispersed as domains in a continuous PP phase. This is the stage where ENR is present in the intra-spherulitic structure of PP (Figure 3). In Figure 7(c), the ENR phase started to enlarge its size and formed bigger ENR domains in PP matrix. The structure was slightly in between proposed structure of 50/50 PP/ENR and 30/70 PP/ENR in Figure 3. In addition, there were smaller PP domains (PP particles) situated in ENR phases as depicted in 8(c). It clearly has shown the condition where occlusions of ENR

phase started to form as the concentration of rubber matrix higher than 50 wt% in the blend; with the presence of PP particulates trapped in the ENR regions.

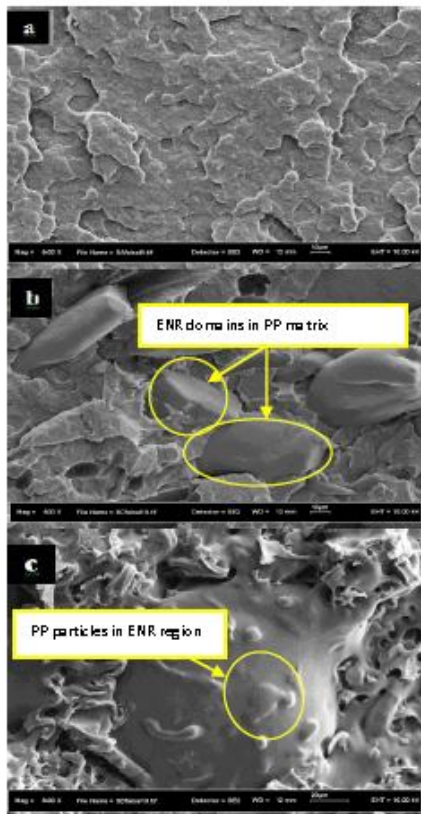


Figure 7 Scanning electron micrograph of (a) unfilled PP, (b) 70/30 PP/ENR and (c) 40/60 PP/ENR at magnification of 500x

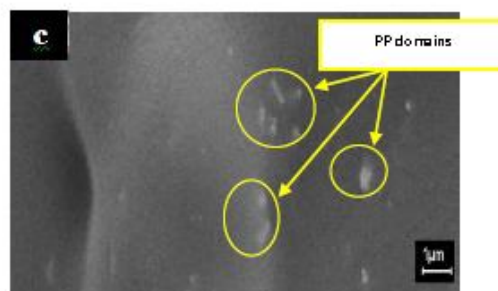
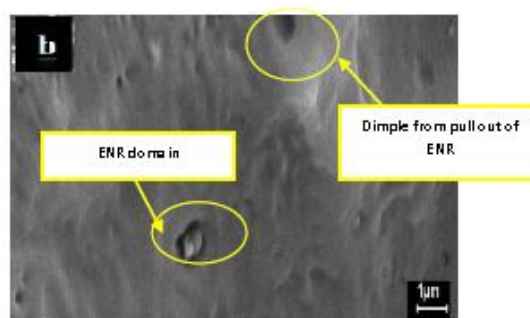
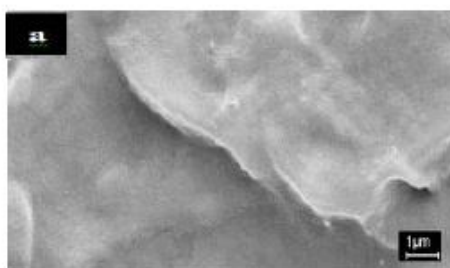


Figure 8 Scanning electron micrograph of (a) unfilled PP, (b) 70/30 PP/ENR and (c) 40/60 PP/ENR at magnification of 5000x

IV. CONCLUSIONS

As a conclusion, it was found that the thermoplastic vulcanizates of PP/ENR at high rubber content shows improvement in its toughness and flexibility. The elongation at break values increases as the rubber content increased in the blend. On the other hand, the addition of rubber content increases impact strength at more than 50 wt % into the thermoplastics matrix. It was clearly proven when 40/60 PP/ENR blend showed increment of impact strength to 2.55 J/m if compared to 1.63 J/m in pure PP. However, the addition of rubber content lowers the tensile strength, Young's modulus and hardness of the blend due to the reduction in PP. These properties are highly contributed by the rigidity of PP chains and ability of it to be strain-crystallized. In contrast, the presence of crosslinking in rubber matrix of 40/60 PP/ENR imparted elastic behaviour and decreased the Young's modulus of PP due to rubber-like properties introduced by the ENR.

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